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SYNTHESIS, OPTIMIZATION, AND POTENTIAL APPLICATION IN EPOXY-BASED COATINGS OF HYDROPHOBIC ACRYLATES

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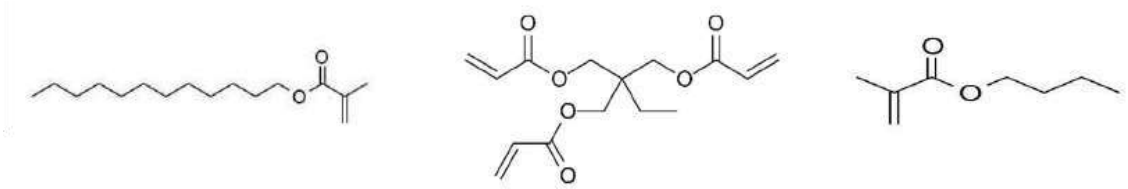
ABSTRACT:

Acrylates are common monomers in polymer chemistry because they can quickly polymerise and have properties that can be changed. Acrylates are the esters, salts and conjugate bases of acrylic acid with its derivatives [1]. Acrylates and methacrylates are esters that come from acrylic and methacrylic acid, respectively. Their molecular structure has a vinyl (-C=C-) group, which makes polymerisation happen quickly through free radical mechanisms. This study examined the hydrophobic properties of two distinct long-chain alkyl methacrylates: decyl methacrylate and octyl methacrylate. This study synthesises and optimises octyl methacrylate (OMA) and decyl methacrylate (DMA), two long-chain alkyl methacrylates, through Fischer esterification of the respective alcohols with methacrylic acid in a nitrogen atmosphere. We changed the ratios of the reactants, the concentration of the catalyst, and the reaction time to get a better yield and purity. We used Fourier Transform Infrared Spectroscopy (FTIR), Thin Layer Chromatography (TLC), and Proton Nuclear Magnetic Resonance (^{1}H-NMR) to figure out what the monomers we made were. OMA was somewhat unstable and had proton irregularities, but DMA was stable and had good structural integrity, as shown by the characteristic acrylate and alkyl chain peaks. DMA was added to epoxy resin matrices to make them less likely to absorb water. We tested the coatings by looking at the water contact angle (WCA). The results showed a big change, going from about 67° for plain epoxy to 88° for DMA-modified coatings. These findings indicate that DMA effectively reduces water repellency on surfaces, implying its potential as an environmentally friendly, high-performance additive for protective coatings in moisture-prone areas.

Keywords Octyl methacrylate (OMA), decyl methacrylate (DMA), hydrophobic, water contact angle, automotive, and architectural coatings.

INTRODUCTION:

Acrylate monomer, which usually comprises of esters which contains vinyl groups, that is two carbon atoms that are double bonded to each other and directly attached to the carbonyl carbon of the ester group. Often, acrylate refers to esters of acrylic acid, the most common member being methyl acrylate [Kali, 2007]. Their excellent durability, clarity, weather resistance, and ease of polymerization make them especially valuable in industrial and commercial applications. Among the most well- known methacrylate's is methyl methacrylate (MMA), which is the principal monomer used to produce polymethyl methacrylate (PMMA), commonly known as acrylic or Plexiglas. [Jalilian, 2016].



Dodecyl methacrylate Trimethylolpropane Tri acrylate Butyl Methacrylate

Fig 1.1: Structure of a few acrylates

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Several products exemplify the use of acrylates in water-repellent architectural coatings. HDP Water-Repellent Coating is an exterior, 100% acrylate coating with excellent water repellent properties to resist Mold and mildew growth resulting in less dirt pick up and cleaner appearance. Available in standard colours as well as customer colours, this coating is used to paint acrylic based textured finishes, masonry, stucco, wood, or primed metal [6]. The hydrophobicity of acrylates increases with the alkyl chain length, making octyl and decyl methacrylates promising candidates for advanced coating formulations. This study aimed to synthesize and optimize OMA and DMA, evaluate their stability and purity, and investigate their influence on the hydrophobicity of epoxy coatings.

MATERIALS AND METHODS

2.1 Chemicals

Octyl alcohol (OA), decyl alcohol (DA), methacrylic acid (MA), concentrated sulfuric acid, ethyl acetate, n-hexane, sodium bicarbonate, sodium chloride, sodium sulfate, diethyl ether, and epoxy resin were utilized in their received form.

2.2 Synthesis of Octyl Methacrylate (OMA)

Octyl Methacrylate (OMA) is synthesized using Fischer esterification of octyl alcohol and methacrylic acid.

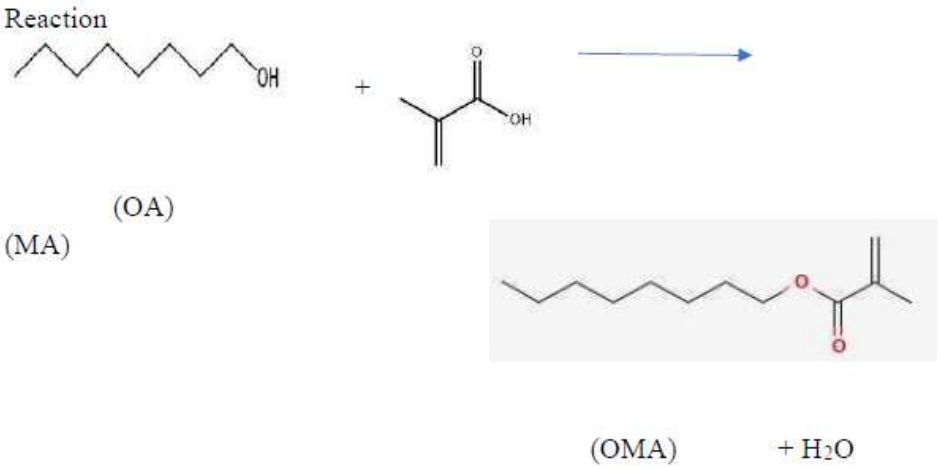

Fig 2.4: Synthesis of octyl methacrylate

2.3 Synthesis of Decyl Methacrylate (DMA)

Decyl Methacrylate (DMA) is synthesized using Fischer esterification of decyl alcohol and methacrylic acid.[16]

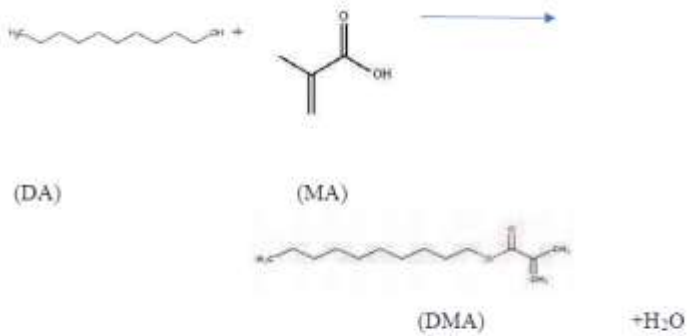

Fig 2.3: synthesis of decyl methacrylate

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RESULTS AND DISCUSSION:

The experimental study aimed to synthesize, characterize, and evaluate hydrophobic acrylate monomers, specifically octyl methacrylate (OMA) and decyl methacrylate (DMA) and their subsequent application in epoxy resin coatings.

3.1 Synthesis of Octyl methacrylate

Octyl methacrylate made azeotropic mixture with ethyl acetate while distilling at 1:1.1 concentration of methacrylic acid to octyl alcohol, the presence of octyl alcohol was observed in Thin layer Chromatography (TLC)

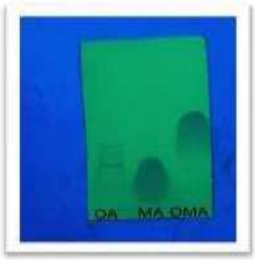

Fig .3.1: TLC of octyl methacrylate

After column chromatography of octyl methacrylate, it was noted that the fraction obtained during distillation produced no residual product (OMA). This made us think that OMA and the solvent (ethyl acetate: n-hexane) form an azeotropic mixture. We were able to get the desired product OMA without using column chromatography by using preparative TLC [1], as shown in Fig. 3.1.

However, it was noted that the time it took to get Octyl methacrylate caused a solid-like structure to form that turned pale yellow, as shown in Fig. 3.2. In addition, while characterising octyl methacrylate, two more protons were found in the monomer, but their nature is still unknown. This makes octyl methacrylate unsuitable for use in the coating, so more research is needed to learn more about its properties.

Fig 3.2 octyl methacrylate

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Batch	Concentration of reactants OA:MA	Reaction condition	Remarks
B1	1:1.1	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	product formed azeotropic mixture with Ethyl acetate while distilling
B2	1:1.1	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	product formed azeotropic mixture with Ethyl acetate while distilling
B3	**1:1.1**	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	% of ethyl acetate was reduced ,product obtained but OA and MA was found in it
B4	**1:1.5**	Reaction was continuously stirred for 48 h with 5-6 drops of catalyst in room temperature	% of ethyl acetate reduced ,product obtained but OA and MA was found
B5	**1:1.5**	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	Workup done Product turned pale yellow over the period of time
B6	**1:1.5**	Reaction was continuously stirred for 48 h with 4 drops of catalyst in room temperature	Workup done Product turned pale yellow over the period of time
B7	**1:1.5**	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	Product obtained by preparative TLC, but formed solid like structure over the period of time at room temperature
B8	**1:1.1**	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	Product obtained by preparative TLC, but formed solid like structure over the period of time at room temperature

3.2 Synthesis of Octyl methacrylate

To make decyl methacrylate (DMA), you need a two-neck round-bottom flask and a 1:1 mixture of decyl alcohol and methacrylic acid. 1. Next, a small amount of sulphuric acid was added. Seal the round-bottom flask and keep it for 48 hours while stirring it constantly. Conditions for the Reaction

1. in a nitrogen-rich environment.

2. at room temperature. Use column chromatography to clean up the compound that you made. Put the compound you got in a place where it won't react with anything.

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Table 3.2 Result of decyl methacrylate

Batch	Concentration of reactants DA:MA	Reaction condition	Yield	Remarks
B2	1:1.1	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	16.6%	Product turned pale yellow MA, H2so4 present
B3	1:1.1	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	60 %	DA,MA,H2SO4 present
B4	1:1.5	Reaction was continuously stirred for 48 h with 5-6 drops of catalyst in room temperature	52%	DA,MA,H2SO4 present
B5	1:1.5	Reaction was continuously stirred for 48 h with 3-4 drops of catalyst in room temperature	26%	bicarbonate treatment pure product obtained
B6	1:2	Reaction was continuously stirred for 48 h with 4 drops of catalyst in room temperature	33%	increase in concentration of MA
B7	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	30 %	Doubled the amount of reactants
B8	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	30 %	doubled the amount of reactants
B9	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	-	no product obtained due to chemical contamination
B10	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	21%	low yield obtained
B11	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	21%	Low yield obtained
B12	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	34.3%	workup was done after a long time
B13	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	30%	workup was done after a long time
B14	1:2	Reaction was continuously stirred for 48 h with 5 drops of catalyst in room temperature	17%	H_2SO_4 found
B15	1:2	Reaction was continuously stirred for 48 h with 7 drops of catalyst in room temperature	-	no product obtained due to human error
B16	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	38.5%	mild H_2SO_4 was found

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B17	1:2	Reaction was continuously stirred for 48 h with 7 drops of catalyst in room temperature	51.5%	mild H_2SO_4 was found
B18	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	44.6%	increase in yield
B19	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	48%	increase in yield
B20	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	56%	increase in yield
B21	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	56%	increase in yield
B22	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	50.6%	mild H_2SO_4 was found
B23	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	50.6%	increase in yield
B24	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	49%	increase in yield
B25	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	57.1%	increase in yield
B26	1:2	Reaction was continuously stirred for 72 h with 7 drops of catalyst in room temperature	46.3%	pure compound obtained
B27	1:2	Reaction was continuously stirred for 96 h with 7 drops of catalyst in room temperature	56 %	DA alcohol was found

Optimization of Synthesis Parameters

Fischer esterification, a condensation reaction between the long-chain alcohol and methacrylic acid, was used to make both OMA and DMA. Sulphuric acid acted as a catalyst in a nitrogen atmosphere. To get higher yields and purity, reaction parameters like the ratio of reactants, the amount of catalyst, and the length of time were changed in a systematic way. For OMA, molar ratios of 1:1.1 initially resulted in low conversion because ethyl acetate formed an azeotrope during distillation. When the concentration of methacrylic acid was raised to 1:1.5, the recovery of the product improved, but the product began to turn yellow and partially solidify over time, which suggested that it was unstable.

DMA synthesis, on the other hand, was stable and could be repeated. Reactions done with a 1:2 molar ratio and 7 drops of sulphuric acid for 72 hours made a product that was up to 56% pure. Residual alcohol and acid were produced when the catalyst loadings were lower or the reaction times were

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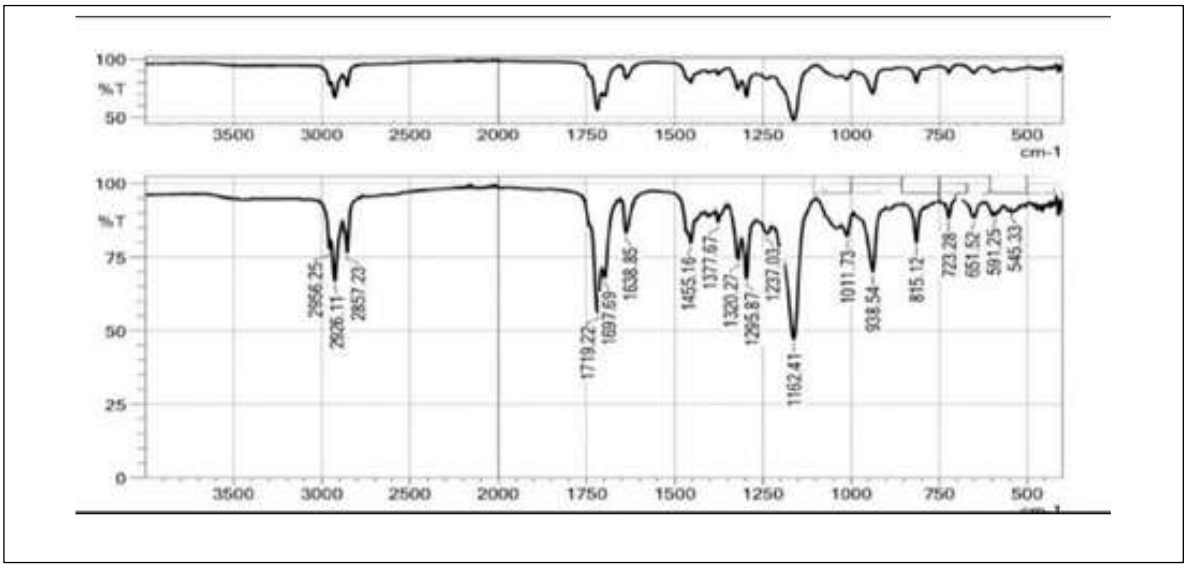
shorter. The longer alkyl chain of decyl alcohol made the reaction slower by adding steric hindrance, but it also made the final product more stable and hydrophobic. A long reaction time made sure that the esterification was complete and that the DMA synthesis could be improved for large-scale, repeatable production.

3.2 Thin Layer Chromatography (TLC) Analysis

TLC analysis was very important for keeping track of progress. For OMA, plates (silica gel, ethyl acetate: hexane 3:7) showed several spots, which showed that the esterification was not complete and there was still octyl alcohol. After preparative TLC cleaned it up, there was only one spot, but the fact that the product changed over time suggested that side reactions were happening. On the other hand, DMA batches from B20 onwards showed a single sharp spot with a higher Rf, which means they were pure esters. The results matched the lack of –OH peaks in FTIR spectra, which proved that the conversion was complete.

3.3 FTIR Spectral Characterization

FTIR spectra validated the formation of esters. Both OMA and DMA exhibited strong peaks at 1640 cm^{-1} (C=C stretch, acrylate) and 1720 cm^{-1} (C=O stretch, ester). The O–H stretch (3200–3600 cm^{-1}) was missing, but the prominent alkyl chain C–H stretches were at 2926 and 2857 cm^{-1}. Figure 3.3 shows the OMA spectrum, and Figure 3.4 shows the DMA spectrum. The OMA spectrum showed small shoulders near 3500 cm^{-1}, which meant that there were still some alcohol molecules that hadn't reacted. DMA, on the other hand, had sharper, cleaner peaks, which meant that the reaction was complete. The clear separation of the bands suggested that the molecules were more ordered, which is in line with the longer decyl chain.



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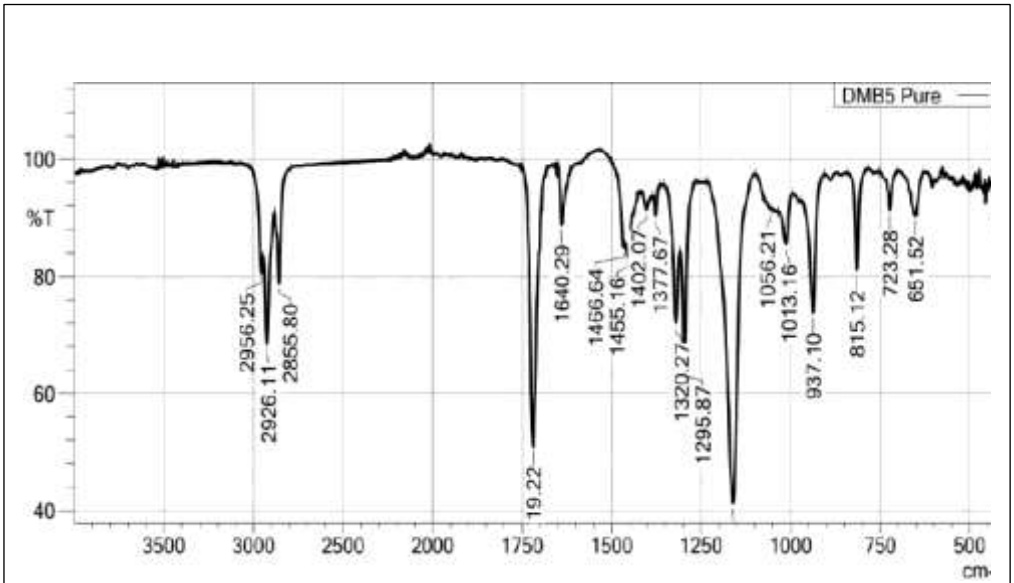

Fig 3.4 FTIR of decyl methacrylate

3.4 Proton Nuclear Magnetic Resonance (^{1}H-NMR) Analysis

^{1}H-NMR spectra (Figures 3.5 and 3.6) provided precise structural confirmation. OMA showed vinyl proton signals at δ 6.10 and 5.55, methylene at δ 4.14, and long-chain protons between δ 1.28–0.88. Extra peaks suggested partial polymerization or solvent residues. DMA displayed δ 6.05 (d,1H), 5.48 (q,1H), 4.09 (t,2H), 1.95 (s,3H), 1.26 (m,14H), and 0.84 (t,3H), matching literature data. No unexpected signals were detected, confirming structural purity and stability.

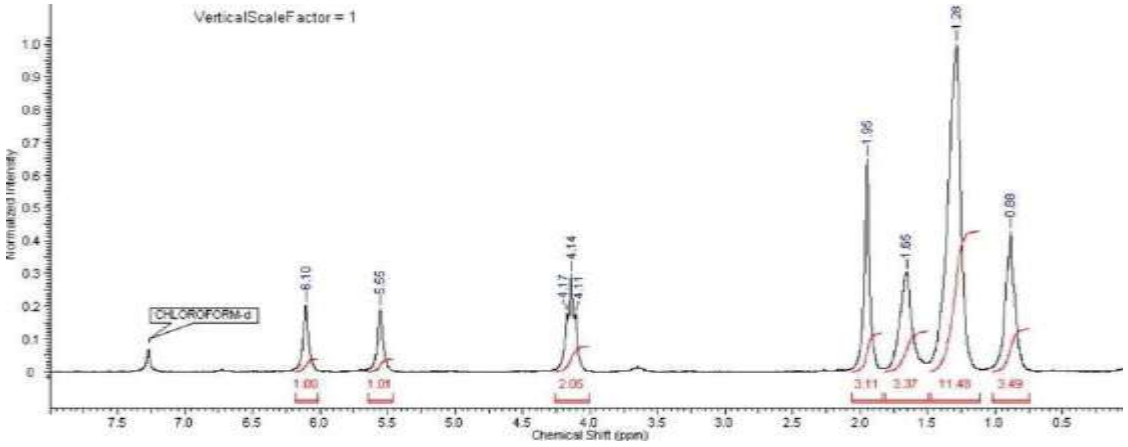

Fig.3.5: NMR for octyl methacrylate

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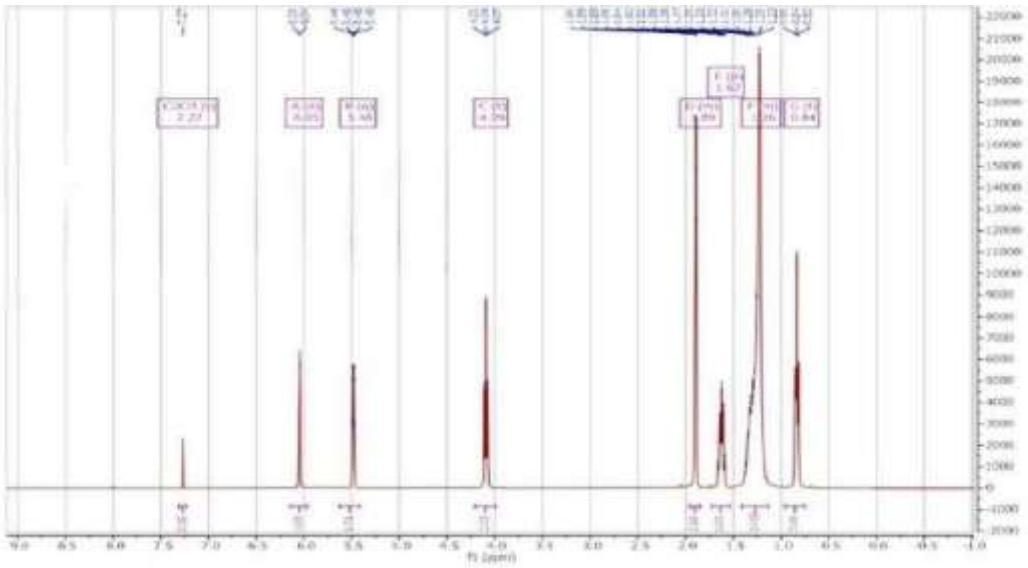

Fig.3.6: NMR for DMA

3.5 Comparative Evaluation of OMA and DMA

Comparatively, OMA demonstrated lesser stability and higher impurity levels, while DMA offered superior purity and consistent hydrophobicity. The extra methylene units in DMA contributed to stronger van der Waals interactions, enhancing surface hydrophobicity and reducing surface energy. The solidification and discoloration of OMA arose from self-polymerization due to unreacted methacrylic acid traces. DMA's higher molecular weight and chain length prevented similar degradation, establishing it as the preferred hydrophobic monomer.

3.6 Adding DMA to Epoxy Resin Coatings

To make the epoxy resin matrices more water-resistant, DMA was added to them (0.5–2 wt. %). Ultra-sonication made the dispersion uniform, and then it was cured at 70°C for 24 hours. Figure 5 shows a diagram of how to prepare the coating. Coatings that had been modified with DMA had smooth, clear surfaces that stuck well to other surfaces. The alkyl chains that are hydrophobic pointed outward, creating a surface with low energy that kept water away.

3.7 Measurements of the Water Contact Angle (WCA)

Figure 3.6 shows how to measure WCA. Neat epoxy coatings had contact angles of about 67°, which means that they were only partially wet. Adding 2 wt.% DMA raised the angle to about 88°, which means that the material is very hydrophobic. The results are shown in Table 3.2. A higher contact angle means less surface energy and better water resistance. This finding is consistent with previous studies indicating that long-chain acrylates enhance surface hydrophobicity (Cheng et al., 2024; Su & Wang, 2023).

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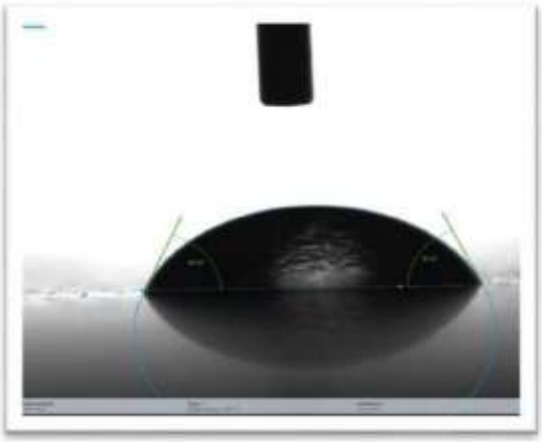

Fig.3.7: WCA for epoxy resin without decyl methacrylate

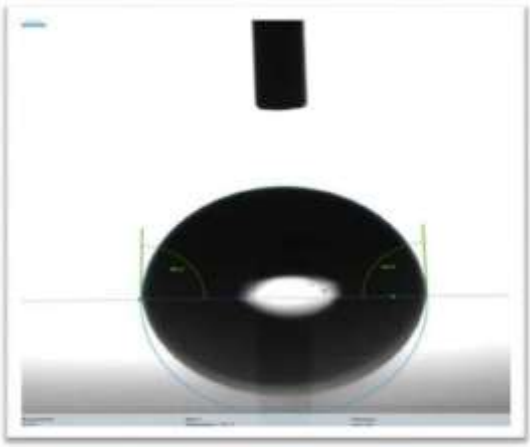

Fig. 3.8: WCA for epoxy resin with decyl methacrylate

3.8 Mechanistic Interpretation and Morphological Behaviour

Molecular self-organization is what makes the material more hydrophobic. DMA's decyl chains move towards the air-polymer interface during curing, creating a hydrophobic outer layer. This surface orientation reduces polar interactions, which helps water droplets keep their round shape. Morphologically, optimised batches (DMEP3–DMEP5) had smoother films with fewer flaws, while early formulations had uneven spreading. The films that came out of this process had both hydrophobicity and clarity, which are both important for industrial uses.

3.9 Relationship between structure and property

The length of the alkyl chain has a big effect on how hydrophobic methacrylate-based coatings are. The decyl group has a longer hydrocarbon chain than the octyl group, which makes the polar part of surface energy lower more effectively. This is why the contact angle goes up by 21° when you change the epoxy to DMA-modified resin. The findings validate the hypothesis that elongated nonpolar moieties improve hydrophobic efficacy while maintaining mechanical integrity.

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CONCLUSION:

Adding decyl methacrylate to an epoxy resin matrix made the coating more water-repellent, as shown by higher water contact angle (WCA) measurements. This means that decyl methacrylate is a good additive for making epoxy-based coatings more water-resistant, especially in marine or moisture-sensitive applications.

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